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THE PROPERTIES OF LONGLEAF PINE OLEORESIN AS AFFECTED BY TREE CHARACTERISTICS AND MANAGEMENT PRACTICES

by

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FOREWORD

The gum naval stores industry is now entering the most highly competitive period in its long history. Steadily mounting competition from highly organized wood naval stores enterprises has sharpened the need for greater efficiency in the traditional methods of collecting oleoresin for the production of gum naval stores.

One focal point of naval stores research has been the reduction of the forty-odd trips that are customarily made to each tree per year for a yield of 8 to 10 pounds of gum. Treatment of the fresh streak with sulphuric acid to prolong the flow of gum now makes it possible to halve the frequency of chipping and to save 16 of the usual 32 trips made each season for this purpose.

Another possibility is to reduce the number of trips made in collecting the gum by increasing the size of the cup on each tree. This report of a study conducted in 1935 is published at this time because of its bearing on the problems that might be connected with the use of larger cups or with various other modifications of turpentining methods to improve their efficiency.

The results have not been published heretofore, though they have been used as a basis for various recommendations on turpentining practices.

Carl E. Ostrom

THE PROPERTIES OF LONGLEAF PINE OLEORESIN AS AFFECTED BY TREE CHARACTERISTICS AND MANAGEMENT PRACTICES

by

C. K. Clark[1] and J. G. Osborne[2]

INTRODUCTION

There are numerous factors involved in the production of naval stores which conceivably might affect the grades of rosin produced and the yield of turpentine obtained, and which consequently would have an important bearing on the economics of the industry. It is important, therefore, that the turpentine operator be familiar with such factors and with their effect on rosin grade and turpentine yield.

Accordingly, a study was undertaken jointly by the Southern Forest Experiment Station of the United States Forest Service and the Naval Stores Station of the Bureau of Agricultural and Industrial Chemistry, with the object of evaluating the effect of tree characteristics and certain management practices on the grades of rosin and the percentage of turpentine obtained from longleaf pine gum and scrape.[3]

This study was carried out on 99 longleaf pines in the Olustee Experimental Forest of the Southeastern Forest Experiment Station near Olustee, Florida. The trees were about 40 years of age, with diameters ranging from 7 to 13 inches at breast height, and showed growth rates ranging from 4 to 15 rings in the last radial inch. The width of all faces was one third the tree circumference, and ½x½-inch streaks were applied at weekly intervals. Initial face heights of from 3 to 32 inches above the gutter were provided. Standard aluminum cups and gutters were used.

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[3] Henceforth in this discussion, that part of the total gum yield which collects in the cup (commonly called "dip") will be referred to as "gum." The portion which remains as a hard coating on the face of the tree will be called "scrape."

The tree factors studied were diameter, growth rate, and inherent yielding capacity. The management practices included varying the average face height[4] from 8 to 36 inches, and a comparison of weekly versus monthly dipping. The test was run from July to November, during which time 16 streaks were applied and 4 monthly and 16 weekly dippings were made.

The selection, measurement, and working of the trees were carried out by the Forest Service, while the collection, storage, and distillation of gum and the examination of the products were done by the Bureau of Agricultural and Industrial Chemistry. Both agencies collaborated in the analysis and interpretation of the data.

FIELD AND LABORATORY METHODS

One hundred trees were selected[5] on a four-acre tract in the Olustee Experimental Forest, and careful measurements were made of diameter at breast height (4½ feet above ground) and the ratio of crown length to total height. Growth rate, expressed as the number of rings in the outer inch of wood, was determined from increment borer cores. By random selection, original face heights of from 3 to 32 inches were assigned to the trees, with the stipulation that face heights be distributed as equally as possible among diameter classes. Individual trees were then assigned at random to the weekly or monthly dipping group, with distribution equal among diameter and face-height classes.

In applying faces, the rough bark was removed from the part of the tree to be chipped, and face and streak dimensions were marked with crayon to insure that chipping would be done to specification. Faces of various original heights were obtained by chipping the initial streak at the desired height above the gutter and removing the intervening wood with a hogal.

4/ By average height of face is meant the mean distance between the height of the fresh streak above the gutter at the start and that at the conclusion of the test.

5/ One tree subsequently died.

At each dipping the gum from each tree was placed in a numbered glass jar and weighed. To prevent oxidation and evaporation during the intervals between dippings, the air in the jar was displaced with carbon dioxide from a cylinder, after which the jar was tightly sealed and stored in a refrigerator. At the conclusion of the field work there had thus accumulated a composite sample of the gum from each tree, while the individual weighings provided a weekly (or monthly) yield record for each tree. All trees were scraped at the end of the season, the scrape from each tree being collected and weighed separately.

Laboratory examination of gum and scrape samples involved distillation to determine water, trash, rosin, and turpentine contents and photometric measurements on the rosin to determine its grade (color index).

The distillation procedure was standardized as closely as possible and was carried out as follows: A weighed amount (600-800 grams) of gum was placed in a two-liter Florence flask which was set in an oil bath maintained at 185±10° C., and was connected to a steam generator and a condenser. As soon as most of the water had distilled out of the sample, as indicated by a gradual decrease in the rate of distillation, steam was admitted at the rate of 5-6 grams per minute until the distillate contained roughly 5 percent of turpentine. The rosin remaining in the still had a temperature of 170-180° C. It was weighed and poured through cotton batting into a special mold to obtain a sample for grading.

As a result of the care exercised in dipping, the trash content of gum samples was too low to affect the weight of rosin appreciably. Scrape, on the other hand, naturally contained chips, bark, and other trash which had to be accounted for in the analysis. This was done by straining the hot rosin from scrape through a 40-mesh wire screen in addition to batting, dissolving the rosin from the trash so collected, and weighing the dried trash.

In calculating gum composition from the distillation results, water content and trash content were calculated on the basis of the weight of crude gum put in the distilling flask. Accordingly, all gum yields were corrected for water content, and henceforth in this discussion the expression "gum yield" refers to actual gum yield corrected for water content.

Turpentine content, on the other hand, was calculated as percentage on pure gum, the weight of pure gum being taken as the sum of the weights of rosin and turpentine produced in the distillation. Thus various samples of gum may be compared on the basis of their turpentine content.

In a preliminary test to determine the reproducibility of distillation results, six samples were taken from a carefully mixed batch of gum and were distilled according to the foregoing procedure. The maximum deviation of turpentine content from the average of the six determinations was 0.40 percent. The conclusion is justified that the results of turpentine distillation are accurate within ±0.5 percent. The grades of rosin made in this test were indistinguishable.

In order to obtain a numerical value for rosin color which could be handled as a variable in the correlation of data, the color of each sample was determined from light-transmission measurements made with a photo-electric colorimeter developed for this purpose by B. A. Brice[6] of the Food and Drug Administration, Washington, D. C. Measurements were made on a 7/8-inch thickness of rosin which had been poured into a glass mold immediately on completion of distillation. The green transmission, G, and the red transmission, R, of the sample were measured by balancing the circuit using blue-green and red filters. The ratio G/R was taken as a measure of chromaticity and the empirical relation G/(R+10), which weighs brightness with chromaticity in good agreement with visual grading, was taken as the color index. The color index corresponding to each standard grade of rosin is shown in table 1.

Table 1.--Photometric rosin grades

Grade	Color index	Grade	Color index
X	0.519	K	0.257
WW	.443	K	.209
WG	.395	H	.152
N	.346	G	.106
M	.304	F	.066

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[6] "A Photoelectric Colorimeter and Its Application to Grading Rosin," by B. A. Brice. This paper was presented before a joint meeting of the Optical Society of America and The American Physical Society in New York, February 24, 1934. Abstract published in Journal of the Optical Society of America, Vol. 24, p. 162 (June 1934).

DATA ANALYSIS

The data obtained on the experimental trees and on their gum and distillation products were analyzed by fitting appropriate multiple regression equations,[7] and, where desirable, the errors of estimate were segregated by frequency of dipping classes and were analyzed. The significance of correlation indexes so obtained was tested by analysis of variance technique.[8] All illustrations given with the following sections show the results of these analyses.

Relationship of Turpentine Content to Face Height, Gum Yield Per Tree, Growth Rate of Tree, and Tree Diameter

A multiple correlation analysis indicated that the turpentine content of gum is significantly related to face height, gum yield per tree, growth rate of tree, and tree diameter, although no single variable exerts a great influence. These relations are shown in figure 1. Black and Thronson[9] report higher turpentine yields from larger trees, and they also state that no relation appears to exist between turpentine yield and the character of the tree crown. Their deductions, however, were based on only a few trees, and the variables were not segregated by a statistical analysis of the data.

It was found in this study that gum from monthly dippings had on the average a higher turpentine content than that from weekly dippings, the difference being 0.45 percent. When tested by its error, this difference was found to be statistically significant.

The indicated difference, 0.45 percent, assumes greater importance when considered in relation to the average percent turpentine in the gum.

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[7] See "Correlation and Machine Calculation," by H. A. Wallace and George W. Snedecor, published by Iowa State College of Agriculture and Mechanic Arts, Ames, Iowa, June 1931.

[8] See "Calculation and Interpretation of Analysis of Variance and Covariance," by George W. Snedecor, published by Collegiate Press, Inc., Ames, Iowa, 1934.

[9] Black and Thronson, "Oleoresin from Individual Trees of Slash and Longleaf Pine," Industrial and Engineering Chemistry 26, p. 66, 1934.

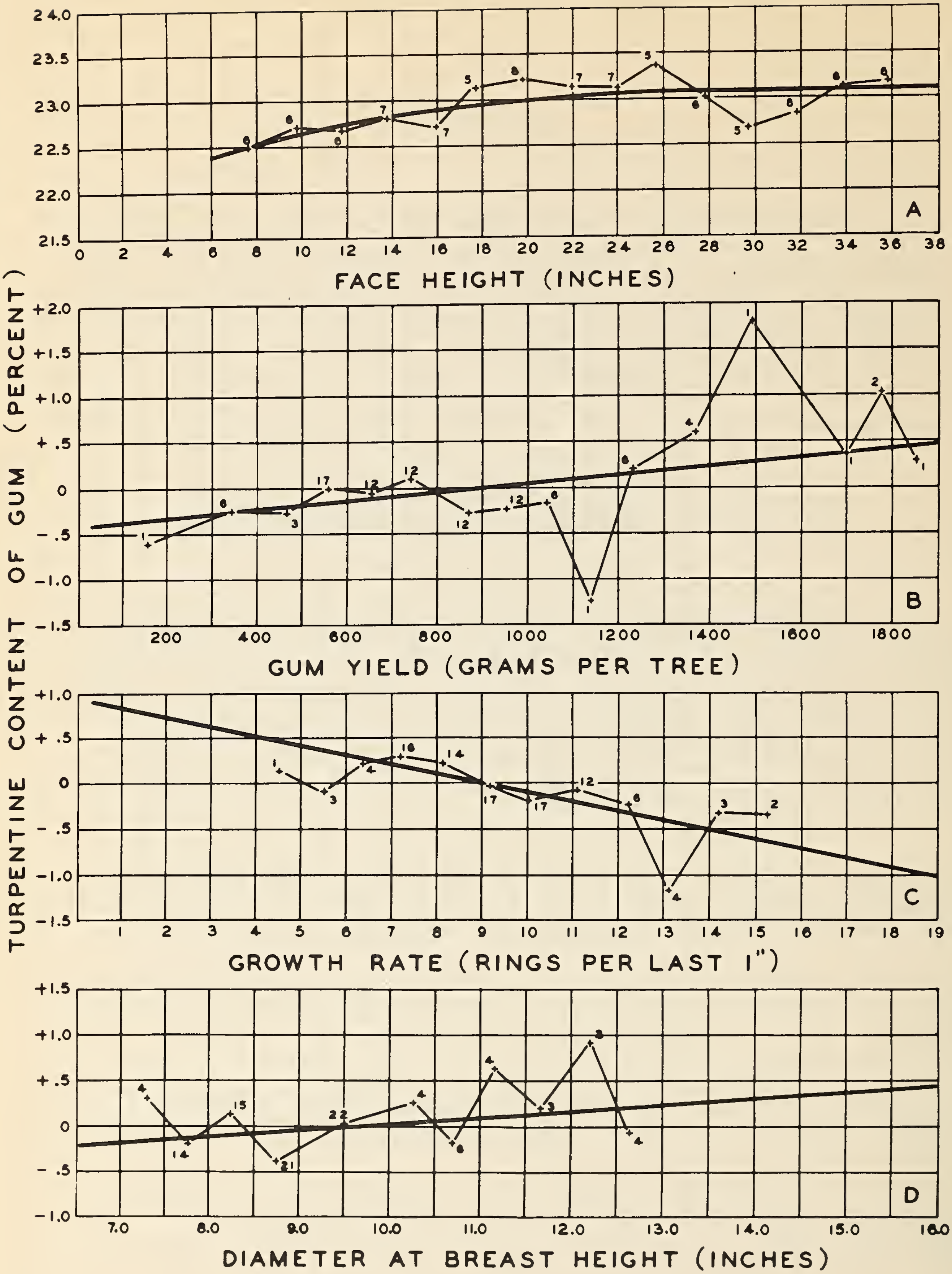

Figure 1.—Turpentine content of longleaf pine gum as correlated with face height, gum yield, growth rate, and diameter at breast height. In (A) turpentine content is read directly according to face height. (B), (C), and (D) show corrections to reading in (A) according to the gum yield, growth rate, and diameter at breast height.

The average turpentine content of the gum from these trees was 22.97 percent. Therefore, dipping monthly resulted in an actual turpentine yield of 0.45/22.97, or 1.96 percent greater than did dipping weekly.

In view of the fact that these findings are at variance with the common belief that frequent dipping results in increased turpentine yield, it becomes necessary to evolve a rational hypothesis which will satisfactorily account for these relations.

In this connection, mention must be made of the experiments conducted in 1923 by V. L. Harper, of the Southern Forest Experiment Station. His unpublished data indicate that very small quantities of pine gum when exposed to the air lose weight by evaporation of turpentine at an appreciable rate for 1½ to 2 days, after which the rate becomes negligible because of the formation of a protective film on the surface of the gum. This period of appreciable evaporation being shorter than either of the two periods tested in the present study, frequency of dipping from this point of view is unimportant.

On the other hand, consideration must be given to the protection against evaporation of turpentine afforded by water in the cup. During a normal turpentine season in this region, rainfall is frequent and water usually stands in the cups. This was especially true in the present instance; rainfall was excessive during the course of the experiment, and the cups were filled with water practically the entire time. Accordingly, when gum was dipped monthly, water was present almost continuously, with the result that a large proportion of the gum yield received protection as soon as it reached the cup. When the gum was dipped weekly, on the other hand, the water was poured out of the cups at each dipping, and unless rain fell shortly after dipping, the succeeding flow was exposed to the air. Since the trees were chipped immediately after dipping, the first surge of gum in the weekly dipped group invariably found a dry cup, while in the monthly dipped group the weekly streaks were chipped nearly always when the cup contained water. This explanation of the effect of water coincides with a long-standing belief among turpentine men. By the same token, it is probable that a similar experiment carried out in an exceptionally dry season would show little or no advantage, so far as turpentine yield is concerned, in either long or short dipping intervals, since one week's flow would be covered and hence protected by the succeeding week's flow.

In this connection it would be well to mention results obtained in a few simple gum exposure tests in which exceptionally long exposure times were used. In these tests, a sample of ordinary commercially produced gum was thoroughly mixed and divided into a number of equal portions as nearly alike as possible. Half of these were distilled to determine the composition of the gum, and the remainder were distilled after being exposed to the weather. In all cases care was exercised to prevent rain water from entering the containers; so the gum was exposed directly without the protection of a layer of water.

It was found that gum exposed in a layer 3/8-inch thick for 30 days had its turpentine content reduced from 20.32 percent to 19.99 percent, which is a loss of 1.6 percent in the turpentine originally present. The grade of rosin dropped from WG to N (one grade).

Another sample exposed in a layer 0.12 inch thick for a period of 41 days had its turpentine content reduced from 23.1 percent to 20.4 percent, which is a loss of 12 percent in the turpentine originally present. The grade dropped from WW to I (five grades).

These findings show clearly that loss of turpentine by evaporation (as well as lowering of rosin grade by oxidation), is dependent upon the proportion of gum surface exposed to the air. Furthermore, the frequency of dipping having been shown to exert little influence on the turpentine yield, it follows that the interval between dippings in a practical operation would be determined largely by such factors as rate of gum flow, size of cups, and the value of gum saved through closer supervision with more frequent dipping.

Grade of Rosin from Gum

From the point of view of the turpentine operator, the importance of improving the grade of rosin he produces varies considerably from time to time, and is greatest, of course, when wide price differentials exist among the various grades. The incentive to produce high grades does exist, however, and therefore it is important that the operator be familiar with the factors which determine the rosin grade. Accordingly, a study was made of the effect of differences in face height on rosin grade, allowance being made for the possible influence of gum yield.

The study showed that low faces on high-yielding timber will produce the highest grades of rosin (fig. 2). To illustrate this effect, the values given in table 2 were calculated from the regression equation on which figure 2 was based.

Thus it can be seen that 38-inch faces (corresponding to working timber 3 years without raising cups) on 29-barrel timber would yield rosin with a market value of $9.52, while 8-inch faces (average height for 1 year's work) on 52-barrel timber would yield rosin with a market value of $11.00, the differential on March 23, 1937, between WW and X being $1.48. On December 6, 1930, the differential was $2.20. The incentive, on this basis alone, for working high-yielding timber and raising cups often (annually) is obvious.

A test was made to determine whether or not the data showed any relation between rosin grade and frequency of dipping. It was found that

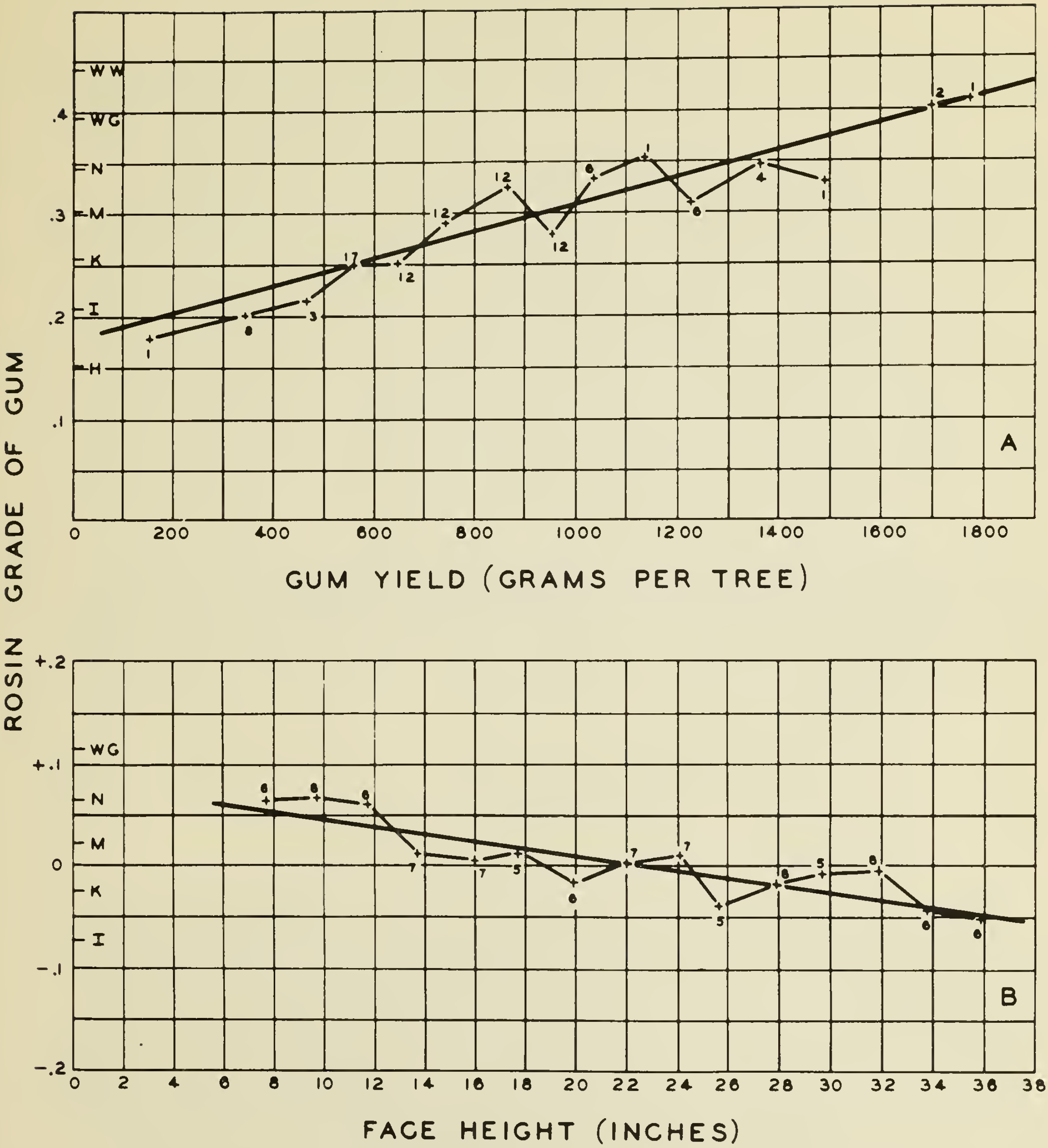

Figure 2.—Rosin grade of gum as correlated with (A) gum yield and (B) face height. Rosin grade is read in (A), with a correction for face height in (B).

monthly dipped trees gave a slightly higher grade of rosin, but a test showed that the difference was not statistically significant. The inference is that frequency of dipping did not influence rosin grade.

Table 2.--Rosin grades corresponding to various yields and face heights

Average face height (Inches)	Gum yield		Estimated rosin grade	
	Grams per tree for 16 streaks	Barrels of spirits per crop[1]	Color index	Grade
8	1,035	29.2	0.360	N
8	1,858	52.4	.468	WW
23	1,035	29.2	.308	M
23	1,858	52.4	.415	WG
38	1,035	29.2	.255	I
38	1,858	52.4	.363	N

[1] It is customary in the industry to express the yield of timber in "barrels per crop," which means the yield of 50-gallon barrels of turpentne (and the corresponding amount of rosin) per 10,000 trees per season. The yields given in grams of gum per 16 streaks were converted to this basis.

Scrape Yield

While flowing from the fresh streak down the turpentine face to the cup, a part of the gum exuded by the tree undergoes chemical and physical changes. Exposure in very thin films results in the evaporation of part of the turpentine, the oxidation of both turpentine and rosin acids, and possibly the isomerization and crystallization of certain resin acids; and the gum so changed solidifies. In this form it is known as "scrape," which may be allowed to accumulate and collected at the end of the season, or it may be "punched" from time to time and collected with the dip. The latter practice is most common, with the result that commercial gum collected after midseason usually contains an increasing proportion of scrape.

Any study of gum yield, especially if it is concerned with the products of distillation, must include a consideration of scrape. When it is distilled, scrape yields considerably less turpentine and a lower grade of rosin than gum. Consequently, a great deal of effort is expended by progressive producers to lower the percentage of gum transformed to scrape. This is the chief reason for raising cups annually.

The relation between scrape yield and face height, gum yield, and tree diameter was established as a basis upon which to determine the value of frequent raising of cups. The results which are of high statistical significance are shown in figure 3.

These relations may be presented in another way in which they may be more directly applicable. It is evident that the scrape which forms on a turpentine face represents a portion of the gum yielded by the tree which did not reach the cup, and that the fraction of the total yield which appears as scrape is dependent upon various tree factors and operating practices. Therefore, it is worth while to calculate the gum equivalent of the scrape obtained and to express the ratio of this value to the amount of gum collected in the cup as a function of the various factors studied. This was done, and the results are shown in figure 4. From these data it is apparent that the fraction of gum going into scrape is large for low-yielding trees, and that it drops rapidly with increasing yield to a point where the yield corresponds to about 30 barrels per crop, after which there is nearly constant ratio.

Figures 3 and 4 reaffirm results published by V. L. Harper in 1928, Southern Forest Experiment Station Notes, that small trees yield a higher proportion of scrape than large trees.

Grade of Rosin from Scrape

On the basis of analyses of 20 samples of scrape, the relation between rosin grade and face height and scrape yield was determined. As can be seen from figure 5, the grade of rosin from scrape rises rapidly with increasing face height, but is practically uninfluenced by scrape yield. This can be explained on the basis of the observation that scrape accumulates to a greater depth on the bottom of the face than toward the top, with the result that the average thickness of scrape is greater on low than on high faces. The truth of the last statement is indicated in figure 3a, which shows that doubling the face height does not double the scrape yield. Hence, the proportion of scrape exposed to the bleaching effect of sunlight is greater on high than on low faces. This certainly is more of academic than practical interest, since the slight advantage of high faces in producing slightly higher grades of scrape is far offset by other harmful effects.

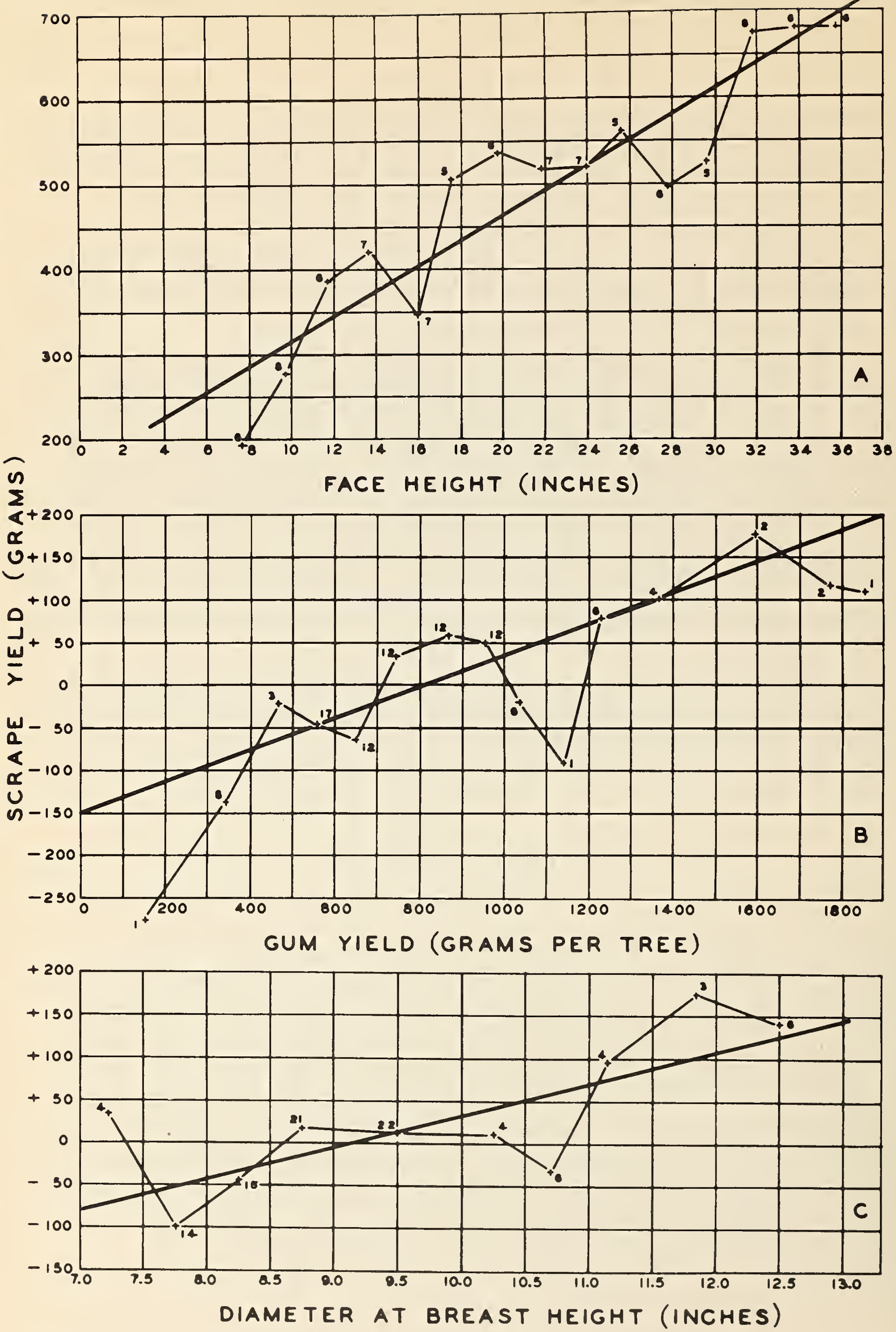

Figure 3.--Scrape yield as correlated with (A) face height, (B) gum yield, and (C) diameter at breast height. Scrape yield is read in (A) with cor-

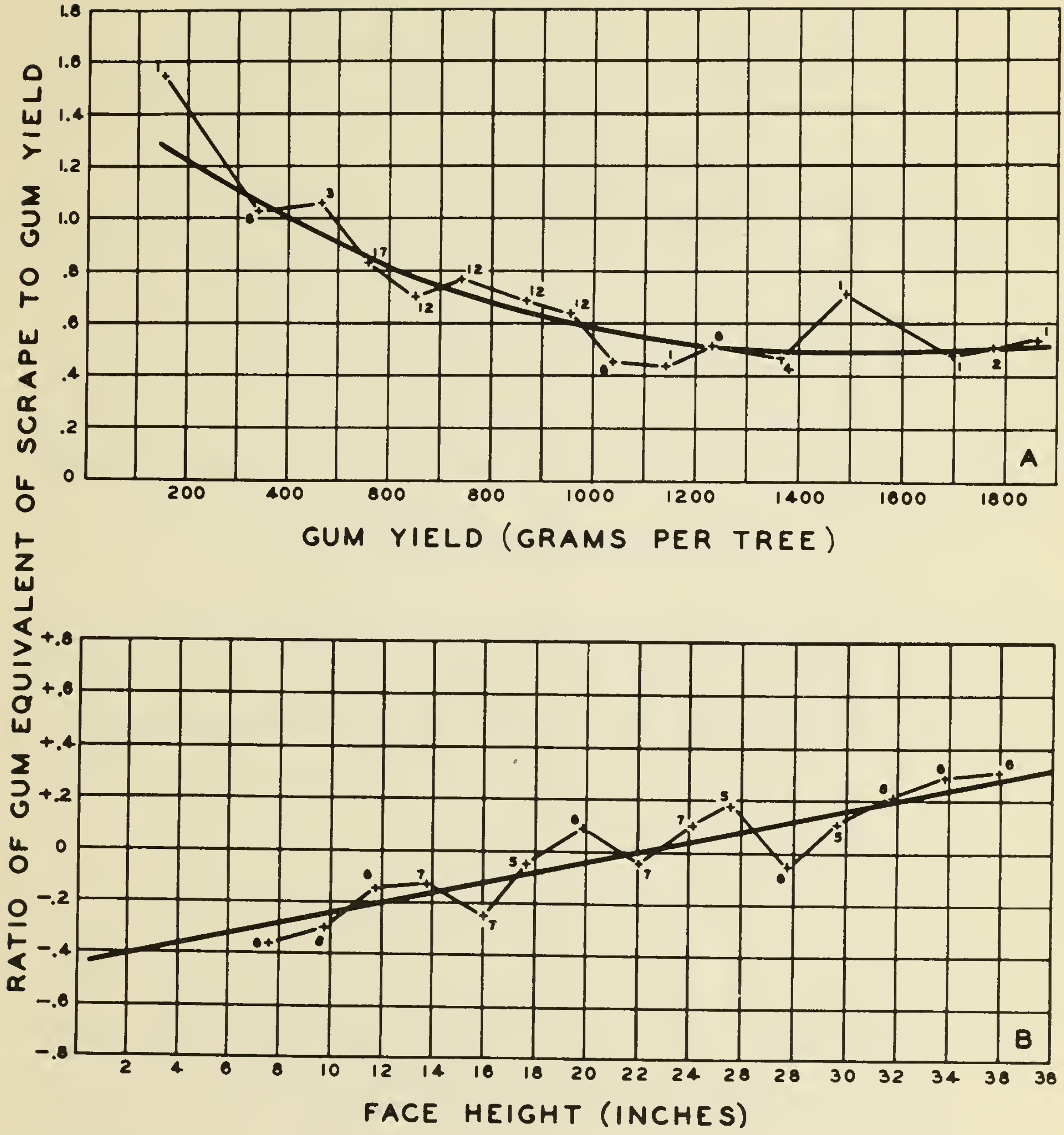

Figure 4.—Ratio of gum equivalent of scrape to gum yield as correlated with (A) gum yield and (B) face height. The ratio is read in (A), with a correction for face height in (B).

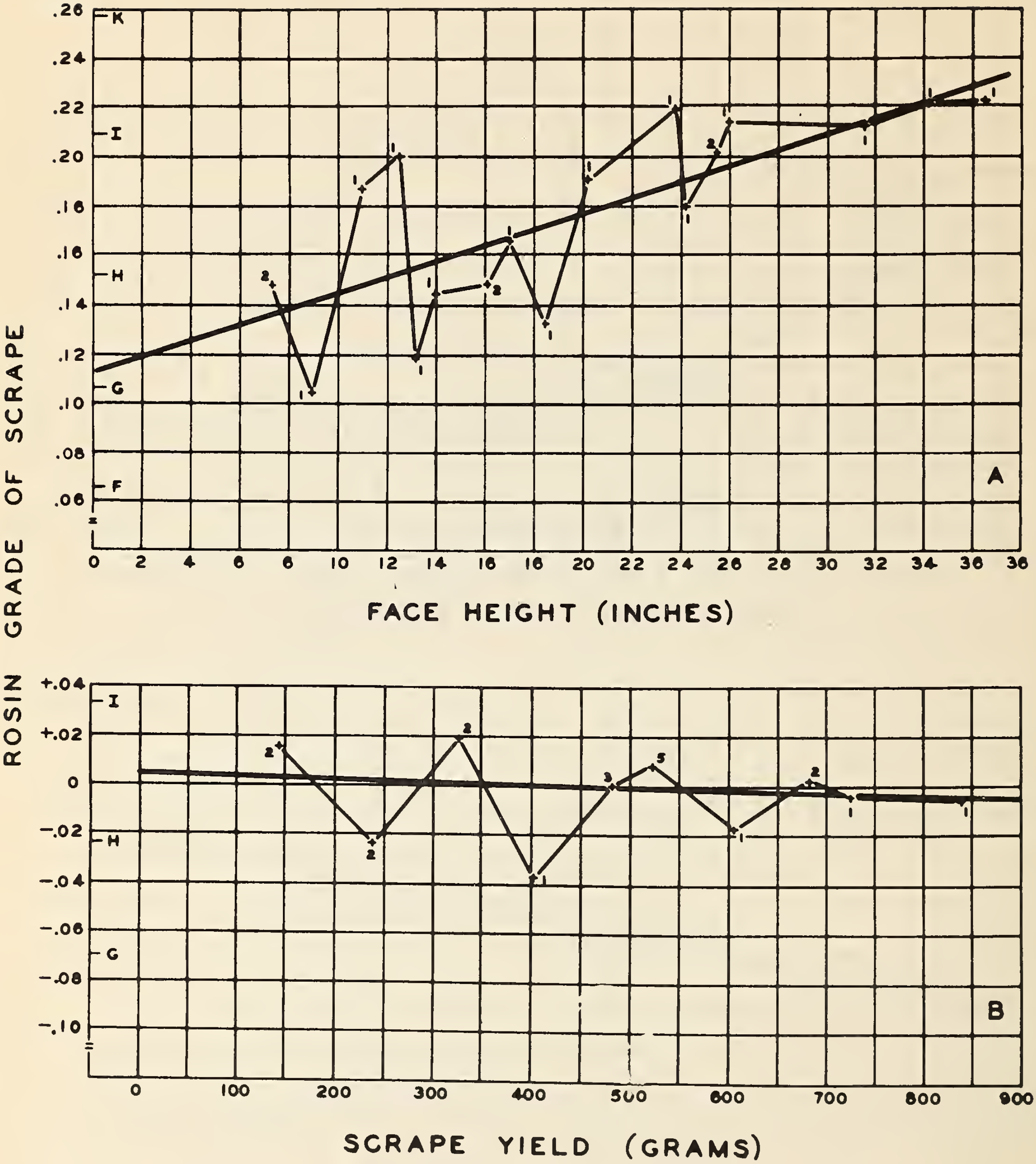

Figure 5.—Rosin grade of scrape as correlated with (A) face height and (B) scrape yield. Rosin grade is read directly in (A) with a correction for scrape yield read from (B).

CONCLUSIONS

1. Turpentine content of gum is influenced somewhat by (a) the height of the turpentine face, (b) the yield of gum, (c) the growth rate of the tree, and (d) the size of the tree (d.b.h.). However, the influence of any one factor is so small as to be of no practical consequence.

2. Turpentine content of gum collected in a rainy season is slightly higher when the gum is dipped monthly than when it is dipped weekly, but the difference is not sufficiently large to be considered in planning dipping schedules.

3. Indications are that no relation exists between turpentine content of gum and inherent gum-yielding capacity of the tree.

4. The grade of rosin produced from gum is strongly correlated with the height of the face and the gum yield. High grades are associated with low faces and high yields.

5. The grade of rosin produced from gum collected in a wet season is uninfluenced by the frequency of dipping.

6. The fraction of the total gum yield which is converted to scrape depends largely on the gum yield and the area of the face. A low proportion of scrape is obtained from low faces on high-yielding trees.

RECOMMENDATIONS

On the basis of this study the following recommendations may be made to the turpentine industry:

1. Frequency of dipping.--Factors other than quality of product, namely, turpentine content of gum and the grade of rosin obtainable, are of consequence in determining the best time interval between dippings. Frequent dipping results in closer supervision, with consequent diminution of gum losses from overflow, leaky gutters,

and cups knocked off the trees by cattle and hogs; hence it is recommended.[10]

2. Raising of cups and gutters.--The value of maintaining short faces is generally recognized in the industry, but this recognition is not based on any quantitative information as to the effects of high and low faces. Raising cups frequently will bring about the following desirable results:

a. The over-all yield of turpentine will be increased through a decrease in the proportion of gum going into scrape.

b. The average grade of rosin produced (from gum and scrape combined) will be raised materially.

c. The amount of rosin going to waste on chips and dross will be materially lessened because of the decreased proportion of scrape (with high trash content).

d. Still-capacity will be increased, and losses of time and material caused by "boil-overs" will be decreased. It was noted in the present study, and it is commonly known in the industry, that gum from high faces has a tendency to foam and "boil high."[11]

3. Working of high-yielding trees.--This procedure is recommended for the following reasons:

a. The most obvious advantage of working high-yielding trees is the greater return per unit of labor outlay.

b. Because a better grade of rosin results from a high proportion of dip and low proportion of scrape, rosin of higher average grade will be produced from the gum of high-yielding trees.

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[10] Editor's Note: In view of recent developments, Carl E. Ostrom, in charge of naval stores research at the Lake City (Florida) Branch of the Southeastern Forest Experiment Station, has this to say: "The question of optimum frequency of dipping was re-opened in 1948 in a pilot-plant study of the cost of dipping average-size and over-size cups. It is quite possible that under present conditions of severe competition for labor and for markets the use of larger cups and longer dipping intervals may have more advantages than disadvantages. The data obtained by Clark and Osborne in the original study will be of particular value in interpreting the results of the present test of the use of larger cups."

[11] See also Department of Agriculture Bulletin 898, Veitch and Grotlisch, "Turpentine," page 3.

c. A higher over-all yield of turpentine will be obtained from gum of high-yielding trees because of its lower proportion of scrape.

d. Less rosin will be lost in still-wastes.

These findings lend added support to the recommendation that no trees under nine inches in diameter at breast height should be worked for turpentine.